Gravimetry

Gravimetry is useful when a quantitative determination of major elemental constituents with high precision and accuracy (better than 0.5–1%) is required. It is often used as a standardization or umpire technique for other quantitative methods.

Principle of Technique

In direct weight loss measurements, samples are weighed before and after treatment, (e.g., heating or rinsing). Precipitation methods involve reaction of analyte with a compound to form a substance of known stoichiometry that can be isolated by filtration and weighed.

Samples

Form. Solids (e.g., metals, ceramics, glasses, polymers) are usually required for direct weight loss measurements. Solutions or dissolved solids and liquids are suitable.

Size. Quantity must be sufficient to contain at least 50 mg of analyte per determination if precipitation methodology is employed.

Preparation. Weight-loss samples are analyzed as received. Dissolution of the sample is required when precipitation methodology is employed.

Limitations

Precipitation methods are slow compared to titration and spectrometric methods, but parallel processing is often possible. A relatively large amount of sample is required. Weight loss is a rather nonselective method.

Estimated Analysis Time

For direct weight-loss measurements, 2 to 4 h is generally needed per analyte. Precipitation methods usually require about 8 h per analyte. High-temperature heating methods usually take about 2 days.

Capabilities of Related Techniques

Titration techniques are usually of equal accuracy and precision. They are more rapid than precipitation methods but operationally are more complicated.

Coulometry is applicable to smaller samples and has comparable accuracy. The various atomic spectroscopy methods may be applicable but could have lower precision and accuracy. Generally, atomic spectroscopy is preferred over gravimetry because of convenience and the capability of measuring nanogram to microgram quantities of material.

Examples of Applications

- Determination of water by weight loss on heating.
- Measurement of metals in organic materials at high concentrations.
- Quantitative determinations of constituents by selective dissolution and weight loss.
- Preparation of oxides of known composition by heating in air.

Standard compilations of relative solubility at 25°C, such as this one for the alkaline earths, are a starting point for the development of selective precipitation procedures.

Calcium compounds	Solubility (moles/L)	Strontium compounds	Solubility (moles/L)	Barium compounds	Solubility (moles/L)
$CaCrO_4 \cdot 2H_2O$	1.0	$Sr(OH)_2 \cdot 8H_2O$	0.084	$Ba(OH)_2 \cdot 8H_2O$	0.27
$Ca(OH)_2$	0.020	$SrCrO_4$	0.0047	BaF_2	0.0069
$CaSO_4 \cdot 2H_2O$	0.015	SrF_2	0.0031	$BaC_2O_4 \cdot 2H_2O$	0.00048
CaF_2	0.00020	$SrSO_4$	0.00082	$BaCO_3$	0.000091
$CaCO_3$	0.00013	$SrC_2O_4 \cdot H_2O$	0.00026	$BaCrO_4$	0.000012
$CaC_2O_4 \cdot H_2O$	0.000049	$SrCO_3$	0.000055	$BaSO_4$	0.000010

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